# PATENT SPECIFICATION

NO DRAWINGS

836,464

Date of filing Complete Specification May 22, 1958.

Application Date July 29, 1957.

No. 23891/57.

Complete Specification Published June 1, 1960.

Index at acceptance:—Class 129, A5.

International Classification:—A23f.

COMPLETE SPECIFICATION

## Method of Producing a Water Soluble Coffee Concentrate

I, RAMON PERECH, citizen of the United States of America, of 915, West End Avenue, New York, State of New York, United States of America, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a new improved method of producing a dry, water soluble coffee concentrate, known by the popular name of Instant Coffee.

All the variations of Instant Coffee available on the market, differ from freshly roasted coffee, owing to the lack of flavour and aroma characteristic of freshly roasted coffee beans.

My method, as described below, assures that the instant coffee, made according to this invention, produces, when dissolved in water, a coffee beverage having the best aroma and flavour.

One familiar with the steps required to produce instant coffee, as it is generally made from roasted coffee beans, understands why all the variations of instant coffee on the market have lost their flavour and aroma, which the coffee beans acquired when the raw coffee beans are roasted, and why the instant coffee on the market quite often has a tang of roasted hay.

It is a fact that the coffee beans acquire their specific coffee flavour and aroma only after the coffee beans are roasted by heating the raw coffee beans to temperatures up to and sometimes in excess of 250° C.

Among the water soluble constituents of raw coffee beans some 70 to 75% of the soluble solids are constituted by protein, caffeine, tannin-chlorogenic acids, sucrose and fats. In the dry state there are approximately 8.62% of sucrose, approximately 9.2% of tannin-chlorogenic compounds, approximately 17% of fats, 1.20 to 1.31% of caffeine, ect., (see "Report of Investigation of Coffee", by Dr. S. C. Prescott, of Massachusetts Institute of Technology, published by the National Coffee Research Association, in New York, N.Y.).

Caffeine, quantitatively, is a minor constituent of coffee beans, yet it is appreciated for its stimulating and diuretic properties, whereas sucrose, tannin-chlorogenic compounds and fats are the important constituents which cause the flavour, aroma, colour—the properties most appreciated by consumers of coffee beverages.

When coffee beans are fully roasted, of approximately 8.62% of the original sucrose only 0.8% remain in the roasted coffee beans. Most of the sucrose appears to disappear in the early roast. Apparently reducing sugars are first formed. These reducing sugars in turn, react rapidly so that the total amount of sugar decreases at first and more slowly throughout the roast. The sugar reactions seem to be dehydration and polymerization reactions. In place of the natural sugars and simply reducing sugars, high molecular water soluble and water insoluble materials are formed. It is believed that much of the special flavour and aroma of roasted coffee beans are due to the reducing sugars and to a volatile oily compound, known as caffeol.

Caffeol is formed as a result of a complex chemical change developed during the roasting of raw coffee beans.

The tannin-chlorogenic compounds are considered constant constituents of coffee beans, and neither the coffee industry nor research institutions have given adequate attention to the importance of the tannin-chlorogenic constituents of coffee beans as a potential source for creating an appropriate flavour in coffee, particularly in instant coffee, which, as many consumers of instant coffee complain, often has a tang of roasted hay.

As a result of experimental research I have discovered that about 9.2% of tannin-chlorogenic compounds in coffee beans, when oxidized, contribute greatly to the improvement

[Price 3s. 6d.]

in the liquoring qualities of a coffee beverage made from instant coffee. Apparently when the tannin-chlorogenic compounds are oxidized in an aqueous solution, they provide the coffee beverage with a very agreeable astringency and also contribute a much better colour to the instant coffee when it is dissolved, properties much appreciated by coffee consumers.

According to the usual method now known in the industry for producing instant coffee, the coffee beans, as available on the market, are roasted and ground, and the water soluble constituents of the roasted and ground coffee beans are extracted by hot water or steam. After the insoluble parts of the ground coffee beans have been separated from the resulting solution, the excess water in the solution is then evaporated. The evaporation of the excess water in the solution is usually effected under vacuum in such wise that there are distilled off the volatile aromatic substances which form the flavour and the aroma of the roasted coffee beans, and the resultant coffee concentrate has lost substantially all traces of flavour and aroma, which characterise roasted coffee beans.

Any one familiar with the steps of processing coffee beans knows that the coffee tree's fruits, also called cherries, which, when ripe, are of deep purple-crimson colour, contain sealed within them the so-called coffee beans. In order to remove the coffee beans from the cherries, they are broken or pulped, and the coffee beans are separated from the thick outer skin and from the mucilagineous substance adhering to the skin.

After the broken outer skin and the mucilagineous substance are separated from the pulped mass, the coffee beans remain enclosed in parchment-like coats and are covered with tiny spermoderms, which, according to their colour, are called silver skins.

In the modern industrial manner of processing the extracted coffee beans, on the plantations, from the moment the beans are extracted from the cherries, they are submitted to several separate crude treatments, including fermentation in water. After fermentation, the beans, still enclosed in their parchment-like enclosures, are dried by exposing them to artificial heat on, for example, barbecues or trays. In order to separate the beans from the dried parchment-like substances, they are then passed through specially constructed equipment, and when dry and broken the very brittle and tiny particles are thereafter winnowed off. Finally the silver skins (spermoderms) on the coffee beans are torn off by friction, and after curing and polishing, the coffee beans have the appearance of the dry coffee beans as they are known on the market. In the course of these treatments some 8 to 10% of the harvested coffee beans are either broken or otherwise damaged and therefore regarded as waste.

As a result of experimental research I have found that it is possible to produce a perfect, water soluble coffee concentrate by extracting the water soluble constituents from disintegrated coffee beans which may be prepared not only from dry processed and even roasted coffee beans, but also from freshly harvested coffee beans from the trees. Where the disintegrated beans are prepared from freshly harvested beans, the beans are first decorticated by mechanical or other means, whereby the beans are separated from the pulpy mass of the coffee trees' cherries, including the outer skins and mucilage, but still remain enclosed in their natural parchment-like coats and covered by their spermoderms.

When the coffee beans are separated from the outer skin and mucilage, some sticky remnants of the mucilage still cling to the coats in which the beans are enclosed. In order to eliminate these remnants of the sticky mucilage, I submit the coffee beans, or more precisely, the parchment-like coats, to an intensive rinsing in water, until the sticky mucilage is liquefied and dissolved in the water in which they are rinsed.

After the water is entirely drained off, I comminute the unfermented coffee beans, together with their parchment-like coats, into a disintegrated homogeneous mass.

When the parchment-like coats are dry, they become very brittle and break off into very tiny light particles, which are easily winnowed off. The unprocessed raw coffee beans, with or without coats, still remain covered with their natural spermoderms, and so they are disintegrated into a homogeneous mass of raw coffee beans.

After the unfermented coffee beans have been disintegrated with or without the coats, I extract by hot water or steam, the water soluble constituents of the comminuted coffee beans, and separate from the obtained solution the water insoluble parts of the beans.

The second and the most important step in my process involves the oxidation of the water soluble constituents of coffee beans, especially their tannin-chlorogenic compounds in aqueous solution, by exposing the solution to the action of oxygen in an oxygeneous atmosphere such as the oxygen of the air. The oxidation is most effectively obtained in the presence of an innocuous inorganic oxidising catalyst. Potassium permanganate is used to particular advantage, with the result that, when the extracted water soluble constituents are oxidized in aqueous medium and converted into instant coffee, the instant coffee acquires a superior aroma and flavour, and an agreeable astringency. The resulting beverage is reddish-brown in colour.

Oxidation is most effectively obtained when the oxidising catalyst is introduced into the

solution, and the solution is exposed for oxidation, under continuous heating for at least one hour, preferably at a temperature of some 15 to 20° C. below the boiling point of water, but not lower than about 30° C., until the solution has acquired the desired degree of astringency as determined by a qualified coffee tester.

During my experimental research I also carried out oxidation of the water soluble constituents of already processed dry, raw coffee beans. I also oxidized the extracted water soluble constituents of already roasted coffee beans in aqueous medium. Oxidation was effected under the conditions specified above in the preceding paragraph. The instant coffee made from previously processed dry coffee beans, as well as from the roasted coffee beans, was of much higher quality than instant coffee made from coffee solids, which had not been oxidized in aqueous solution, but the best instant coffee was made from the water soluble constituents of coffee beans freshly picked from the coffee trees, even when enclosed in their parchment-like coats.

In the extracted water soluble constituents of raw coffee beans is some albumen matter, which, when dissolved in water, and submitted to evaporation under vacuum, causes the solution to foam. According to my process, the aqueous extract, before submitting it to evaporation is heated for at least one hour in contact with an oxygeneous atmosphere and at temperatures approaching the boiling point of water, whereby the albumen matter coagulates and even becomes condensed with other constituents in solution. In this way no foaming, which interferes with evaporation, can take place.

To eliminate from the solution some insoluble particles, especially those formed by coagulation or condensation during exposure of the solution to oxidation under elevated temperatures, I submit the solution to the known steps of filtration and centrifuging and thereafter I submit the solution to evaporation, preferably under vacuum, until the concentrate attains the consistency required for economical spray drying which concentrates the extract.

As a variation in the step of oxidation of the extracted water soluble constituents of coffee beans in aqueous solution, as described above, I do not introduce the catalyst into the solution, but as soon as the insoluble parts of the coffee beans have been separated from the solution, I submit the solution to evaporation under vacuum until the still unoxidized solution is concentrated into the consistency appropriate for spray drying. Only after the unoxidized concentrate is formed do I introduce into the concentrate thus formed an innocuous oxidation catalyst, preferably potassium permanganate, in a proportion of some 0.01 to 0.03% by weight of the tannin-chlorogenic compounds in the concentrate, and expose the concentrate for oxidation in an oxygeneous atmosphere under constant heating at temperatures not lower than 30° C. for at least one hour until, as described above, the concentrate is properly oxidized and has acquired the desired degree of astringency as determined by a qualified coffee taster.

Unlike the usual methods of spray drying, as customarily used in the coffee industry, involving spray drying the coffee concentrate into dry powder under high temperatures, I perform the spray drying in a manner so that the heat utilized for spray drying of the extracted water soluble constituents of raw coffee beans does not cause any substantial chemical changes in the solids of the coffee concentrate. Therefore, the spray drying is performed at as low a temperature as possible. For example, the temperature at the outlet of the spray drying chamber preferably does not exceed 60° C. In this way the powdered coffee solids leave the spray drying chamber chemically unaffected.

An additional and more adequate cooling of the solids inside the spray drying chamber may be accomplished by admitting atmospheric or conditioned air into the spray drying chamber, thus ensuring that the sucrose is not converted into reducing sugar, no caffeol is formed, and the only physical change taking place is that the coffee solids in the concentrate are converted into a powdery state.

Only after production of the powdered coffee extract do I expose it to the action of a high coffee roasting temperature, as usually applied in the industry. Thus, when exposed to the action of heat at a high temperature, the caffeol is formed, and the sucrose becomes converted into reducing sugar, in such wise that the roasted coffee concentrate in powdered form acquires the flavour and aroma of freshly roasted coffee beans.

While the catalyst referred to in the following examples is disclosed as potassium permanganate, it should be understood that other innocuous oxidizing catalysts may be applied to develop the same or kindred qualities in coffee extract. For example, hydrogen peroxide or copper (cupric) oxide has been proved capable of developing substantially the same quality of instant coffee, but copper oxide is not considered acceptable from the standpoint of food technology. On the other hand, hydrogen peroxide, when added to an aqueous solution of coffee extract and submitted to continuous heating at elevated temperature is subject to sudden decomposition.

It is to be understood that raw coffee beans contain approximately 3.5 to 4.42% of inorganic matter, of which approximately 58.48% is potassium monoxide ($K_2O$) and 1.45% is manganic oxide ($Mn_2O_3$). Thus, by introducing minute quantities of potassium permanganate, I introduce no foreign matter, as

potassium as well as manganese are natural constituents of coffee beans (see "Chemische Zusammensetzung der Menschlichen Nahrungs-und Genussmittel", by Dr. Joseph Koenig, vol. 2, page 378, and also "Chemistry of Coffee", by Dr. Ernest Lockhead, of the Coffee Brewing Institute of America, as well as the "Bulletin of French Academy of Science"—1954, pages 1853 and 1854).

The scope of the invention will be fully understood from the description set forth in the following examples:—

EXAMPLE 1

In order to extract the coffee beans from the cherries I break them and separate the coffee beans from the mass of broken skin and mucilage. At this time, the coffee beans are enclosed in parchment-like coats, and some sticky remnants of mucilage cling to the coats. These coats, with the beans still inside, are intensively washed in water until the remnants of sticky mucilage are liquefied and dissolved in water.

After the water has been entirely drained off, I comminute the coffee beans, together with their parchment-like coats, into a homogeneous disintegrated mass.

When the parchment-like coats are dried, they become very brittle and disintegrate into tiny light particles which are easily winnowed off. The raw, unprocessed coffee beans, with or without the coats, unprocessed and unfermented raw coffee beans still remain covered with their natural spermoderms, and so they are disintegrated into a homogeneous mass of raw coffee beans.

When the coffee beans are disintegrated with the coats on them or after the coats have been separated therefrom, I extract by hot water or steam, the water soluble constituents of the comminuted coffee beans and separate from the obtained solution the water insoluble parts of the beans.

After the insoluble parts of the coffee beans have been separated from the solution, I introduce into the solution of the water soluble constituents of the coffee beans an inorganic oxidising catalyst, calculated as approximately 0.01 to 0.3% of the dry weight of the tannin chlorogenic compounds in the solution.

Among various oxidizing catalyst I usually prefer to use potassium permanganate, and expose the solution for oxidation in an oxygeneous atmosphere, preferably to the action of the oxygen of the air, under continuous heating for at least one hour at a temperature below the boiling point of water, but not lower than 30° C. until, as determined by a qualified tester, the solution acquires the desired degree of astringency. The period of time during which the solution is to be exposed to oxidation under continuous heating, and the percentage of the catalyst introduced, may be varied, depending upon the chemical composition of the individual batch and the origin of the coffee beans submitted for process.ng.

To eliminate from the solution some insoluble particles, especially the particles formed by coagulation or condensation during exposure of the solution to oxidation under elevated temperatures, I submit the solution to the known steps of filtration and centrifug.ng. Thereafter I submit the solution to evaporation of the excess water in the solution, preferably under vacuum, until the concentrate attains the consistency appropriate for spray drying the concentrate into a powdery state.

In contrast to the methods customarily used for spray drying a coffee concentrate to form a powder under elevated temperatures, I spray dry the concentrate into a powdery state under the lowest possible temperatures in such a way that heating of the concentrate causes substantially no chemical changes in the concentrate. I obtained very satisfactory results when I spray dried the coffee concentrate at a temperature at the outlet of the spray drying chamber of approximately 60° C., whereby no reducing sugar and no caffeol were formed.

An additional cooling of the solids particles during the spray drying ensures that substantially no chemical changes will take place. I accomplished additional cooling of the solids inside the spray drying chamber by admitting atmospheric or conditioned air directly into the spray drying chamber, and established that no reducing sugars and no caffeol were formed.

Only after the production in powder form of the chemically unchanged coffee extract do I heat the powder at a high temperature, similar to that used for roasting of coffee beans, until the chemical and physical changes, common to roasting of coffee beans, are completed. In this way, most of the sucrose in the powdered coffee extract is changed into reducing sugar caffeol is formed, and there are developed in the roasted powdered coffee extract the flavour and aroma of roasted coffee beans.

By the method of producing instant coffee, as described in this example, the flavour and aroma are developed in the last step of processing and, as the instant coffee does not undergo any further processing and is admitted directly into closed containers, the flavour and aroma which are developed by heating the powdered coffee extract to elevated temperatures remain in the final product.

EXAMPLE 2

I disintegrate a batch of processed raw dry coffee beans, as they are obtainable on the market, into a homogeneous comminuted mass, and extract by water or steam the water soluble constituents of the disintegrated coffee beans. After the insoluble parts of the

beans are separated from the solution, I introduce into the obtained solution of the water soluble constituents of the coffee beans an innocuous inorganic oxidation catalyst, and expose the solution for oxidation to the action of oxygen of the air under continuous heating for at least one hour at temperatures below the boiling point of water, but not lower than 30° C. until, as determined by a qualified coffee tester, the solution is oxidized to the required degree and has acquired the agreeable astringency. After the oxidation is completed, the solution is evaporated to consistency appropriate for spray drying. The concentrate so obtained is spray dried into a powder which is roasted under conditions similar to those described in Example 1.

EXAMPLE 3

I extract the water soluble constituents of already roasted coffee beans by methods of extraction known in the industry. After the insoluble parts of the disintegrated roasted coffee beans have been separated from the solution, I introduce into the solution an innocuous inorganic oxidizing catalyst, preferably potassium permanganate, and expose the solution for oxidation to the action of oxygen of the air, under continuous heating for at least one hour at a temperature not lower than 30° C. until, as determined by a qualified coffee tester, the solution is adequately oxidized and has acquired an agreeable astringency. After the solution is submitted to filtration and evaporation of the excess water in the solution, the resultant concentrate is spray dried into a powder at an elevated temperature customarily used in the coffee industry for spray drying of extracts of coffee beans.

EXAMPLE 4

In a variation of the process described in the foregoing examples for oxidation of the water soluble constituents of the coffee beans in aqueous solution, I do not introduce the oxidising catalyst immediately after the insoluble parts are separated from the solution, but rather first exclude air from the vessel containing the solution, and submit the solution to evaporation under vacuum to remove the excess water until the consistency appropriate for spray drying is attained. Only after such consistency is attained, but while the concentrate is still unoxidised, do I introduce into the concentrate the innocuous oxidising catalyst and expose the resulting concentrate for oxidation for at least one hour in an oxygeneous atmosphere, at temperatures below the boiling point of water, but not lower than 30° C., and finally spray dry the concentrate into a powder and roast the powder in the manner described in the foregoing examples.

EXAMPLE 5

In a variation of the process described in the foregoing examples, I perform the oxidation of the water soluble constituents of the disintegrated coffee beans, before the insoluble parts of the coffee beans are separated from the obtained solution. I introduce an innocuous oxidising catalyst into the solution and expose the solution to the action of the oxygen of the available oxygeneous atmosphere under constant heating for at least one hour at elevated temperatures lower than the boiling point of water until by determination of a qualified coffee tester the solution is adequately oxidised. I then separate the insoluble parts and submit the solution to concentration, spray drying and roasting in the manner described in the foregoing Examples 1, 2 and 4.

The definition "oxygeneous atmosphere" includes also the oxygen of the air.

WHAT I CLAIM IS:—

1. A process for preparing an improved water soluble oxidized coffee extract, which comprises preparing an aqueous extract from disintegrated coffee beans and heating the formed aqueous extract at an elevated temperature below the boiling point of water in contact with an oxygeneous atmosphere for at least one hour and until the water soluble constituents of the coffee are oxidized in the aqueous extract medium.

2. A process for preparing an improved water soluble oxidized coffee extract as claimed in Claim 1, in which the aqueous extract is heated to a temperature higher than the natural point of fermentation of coffee, and water is removed from the oxidized aqueous extract to form a water soluble oxidized coffee extract.

3. A process for preparing an improved water soluble oxidized coffee extract as claimed in Claim 1, in which an innocuous oxidizing catalyst is introduced into the aqueous extract.

4. A process for preparing an improved water soluble oxidized coffee extract as claimed in Claim 2, in which the coffee extract is roasted at coffee roasting temperatures.

5. A process for producing an improved water soluble oxidized coffee extract as claimed in Claim 1, in which the aqueous extract obtained from the disintegrated coffee beans is concentrated to a spray drying consistency and the concentrate is spray dried at temperatures which do not produce substantial chemical changes in the formed unroasted powdered coffee extract.

6. A process for preparing an improved water soluble oxidized coffee extract as claimed in Claim 5, in which the concentrate is spray dried to a temperature not exceeding 60° C.

7. A process for preparing a water soluble

solid oxidized coffee extract consisting in removing the outer skins and mucilaginous layer from the coffee cherries, substantially as they come from the coffee trees, disintegrating the resulting unfermented coffee beans, preparing an aqueous extract from the disintegrated beans, introducing an innocuous oxidising catalyst into the aqueous extract, heating the aqueous extract at an elevated temperature below the boiling point of water in contact with an oxygeneous atmosphere for at least one hour and until water soluble constituents of the coffee are oxidized in the aqueous extract medium, removing water from the oxidized aqueous extract to form a water soluble oxidized coffee extract under conditions and temperatures such that sucrose in the solution is not converted into reducing sugars and caffeol is not formed, and roasting the coffee solids at coffee roasting temperatures.

8. A process for preparing an improved water soluble oxidized coffee extract from disintegrated unroasted coffee beans consisting in preparing an aqueous extract from the disintegrated beans, heating the formed aqueous extract, for at least one hour, at an elevated temperature below the boiling point of water, but higher than the natural fermentation temperatures of raw coffee beans in contact with an oxygeneous atmosphere in presence of an innocuous oxidising catalyst until the water soluble constituents of the coffee beans are oxidized in the aqueous medium, concentrating the aqueous extract to a spray drying consistency, spray drying said extracted concentrate at temperatures such that sucrose in the concentrate is not converted into reducing sugars and caffeol is not formed, and roasting the coffee solids at coffee roasting temperatures.

9. A water soluble coffee concentrate as produced by the process claimed in any of the preceding claims.

10. A water soluble and oxidized coffee extract produced from unfermented coffee by oxidizing an aqueous extract of unfermented coffee in an aqueous medium at elevated tempertures below the boiling point of water in contact with an oxygeneous atmosphere.

11. A water soluble and oxidized coffee concentrate as claimed in Claim 9, in which water has been removed from an aqueous extract of unroasted coffee to produce a water soluble coffee extract at a temperature at which sucrose is not converted to reducing sugar and caffeol is not formed, and the extract has been roasted at coffee roasting temperatures.

RAMON PERECH.

## PROVISIONAL SPECIFICATION

## Method of Producing a Water Soluble Coffee Concentrate

I, RAMON PERECH, citizen of the United States of America, of 915, West End Avenue, New York, State of New York, United States of America, do hereby declare this invention to be described in the following statement:

The present invention relates to a new improved method of production of dry water soluble concentrated dehydrated coffee product known by the popular name of instant coffee.

All the variations of instant coffee available on the market differ from the freshly roasted coffee by lack of flavour and aroma of freshly roasted coffee beans.

Only my new method, as described below, assures that the instant coffee, made according to this my invention, when dissolved in water, produces a coffee beverage with best aroma and fully bodied flavour.

One familiar with the inevitable steps, required for producing instant coffee, as it is generally made, will understand why all the variations of the instant coffee produced to date, are losing the flavour and aroma which the coffee beans acquire when roasted, whereas the produced instant coffee acquires a tang of roasted hay.

It is well known that the coffee beans acquire their specific roasted coffee flavour and aroma only after they were roasted by exposing to high temperature of some 260° to 300° C. or even higher.

The water soluble constituents of green coffee beans consist of protein, caffeine, tannin-chlorogenic acids, sucrose, fats (some 17%), etc. On dry basis there are some 8.62% of sucrose, some 9.2% of tannin compounds, 1.20 to 1.31% of caffeine, etc. (see "Report of Investigation of Coffee", by Dr. S. C. Prescott, of the Massachusetts Institute of Technology).

After all, caffeine is a minor compound of coffee taste, but is highly appreciated for its stimulating and diuretic properties, whereas sucrose, tannin compounds, and fats are the most important constituents the presence of which in coffee beans, produce the flavour, aroma and colour all three properties highly appreciated in coffee drinks.

When coffee beans are duly roasted, from the original 8.62% of sucrose, in roasted coffee beans remain only 0.8% of soluble sucrose, while the other 8% of the original sucrose disappear in the early roast, forming reducing sugar, and much of specific flavour, aroma and colour are due to the reducing sugar. It is explained by the fact that when sucrose ($C_{12}H_{22}O_{11}$) is exposed to a high temperature much higher than the melting point of sucrose (185° C.)—of some 300° C., by polymerisa-

tion, the sucrose becomes reduced and forms higher molecular, reducing sugar compound, which, as stated above, contributes to the development of aroma, flavour and colour of the roasted coffee beans.

It has been also established that flavour and aroma of roasted coffee beans come also from the volatile oily compound, known as caffeol ($C_9H_{16}O$) formed during the roasting of the coffee beans, when a number of complex chemical and physical changes are taking place.

By exposing the coffee beans to high temperature—carbon dioxide is formed, but when the roasted coffee beans are cooled off and are ground for processing into instant coffee, more than 50% of the carbon dioxide are volatilised and when the ground coffee is submitted to further processing, especially during process of evaporation under vacuum, the carbon dioxide is completely eliminated.

Similar to caffeine, tannin compounds are also constant constituents of coffee beans, and it is really amazing that until now neither the coffee industry nor the research did pay the due attention to the importance of the tannin constituents in the coffee beans for creating an appreciable flavour in coffee, particularly in the instant coffee, which, as most of the instant coffee consumers complain, has a tang of roasted hay.

Many years of experimental work proved that, as indicated above, the 9.2% of tannin compounds in the coffee when oxidized in a degree, the oxidized tannin compounds will greatly contribute to the improvement of the liquoring qualities of coffee drinks, since the tannin compounds when oxidized provide the coffee drinks with an agreeable astringent property, and also contribute, that when the resultant instant coffee is dissolved in hot water, it produces a red-brown colour, greatly appreciated in the coffee drinks.

By the usual method of producing instant coffee, the coffee beans are roasted, ground and water soluble constituents are extracted by hot water or steam. Therefore, after the extraction is made, no matter how low the excess water in the resulted solution of the water soluble constituents of the extracted coffee may be, to produce a heavy concentrate the solution has to be submitted to evaporation, mostly under vacuum. Together with the initially evaporated, first of all are distilled the volatile aromatic substances in the solution. Therefore, the flavour and aroma created during roasting of coffee are evaporated and the resultant coffee concentrate remains without any traces of aroma flavour, proper to roasted concentrate.

The new and important step of my this invention is to oxidize the tannin compounds of the coffee beans in solution in an atmosphere of oxygen, on desire in the presence of an oxidizing inorganic catalyst, preferably using as catalyst potassium permanganate ($KMn_2O_4$), so that the instant coffee produced from so oxidized tannin compounds in solution acquires an agreeable astringency and the resultant coffee drink is red-brown in colour.

During my experimental work I also performed the oxidation in the same way of the extracted water soluble constituents of already roasted coffee beans in solution. In spite of the fact that the resulted astringency was lower than the astringency I obtained when I oxidized the solution of water soluble constituents of raw coffee beans, however, I obtained a very acceptable and greatly improved instant coffee product.

This difference in astringency is explained by the fact, that when raw coffee beans are roasted they lose some 50% of their tannin contents.

After oxidation was performed, I concentrate the solution of the water soluble constituents of coffee beans into a consistency required for economical spray drying into a dry powder.

Unlike the usual way of spray drying the concentrated soluble constituents of coffee beans into a powder under elevated temperatures, I perform the spray drying in a way that the applied for spray drying heat does not produce any chemical or physical changes in the solids of the concentrate. I perform the spray drying at low temperatures when the outlet temperature is of some 55 to 60° C., and by this way the dry powdered coffee solids leave the spray drying chamber chemically and physically unaffected.

A more adequate additional cooling of the solids is perfectly accomplished by admitting atmospheric or conditioned air directly into the spray-drying chamber through a side inlet, thus assuring that the sucrose will not be converted into reducing sugar, that no caffeol will be formed, and that the only physical change that will take place is that the solution solids become converted into a dry powder.

Only after the thus chemically unchanged concentrate in dry powdered form is obtained, I expose the same to the action of high degree temperature, until the required for roasting coffee chemical and physical changes are produced. Only then the caffeol and dioxide are formed, and the sucrose becomes converted into reducing sugar, whereby in the formed dry powdered soluble coffee concentrate are formed in the desired degree the coffee flavour and aroma, much superior to any instant coffee produced to date.

As in the instant coffee produced by my method, the flavour and aroma are developed only in the final step of production, when it does not have to undergo any further processing, and it is directly packed into closed containers, the flavour and aroma, developed

during the last step of processing, remain with the final product for good.

As oxidizing catalyst I prefer to use potassium permanganate ($KMn_2O_4$), but other inorganic oxidizing compounds, like hydrogen peroxide, etc., may be used as catalyst.

The raw coffee beans contain some 3.5 to 4.41% of inorganic matters of which some 58.48% is potassium monoxide ($K_2O$) and 1.45% of manganic oxide ($Mn_2O_3$). Thus, by introducing minute quantities of potassium permanganate as catalyst, I introduce no foreign matter, as potassium as well as manganese are natural constituents of the coffee beans.

(See "Chemical Zusammensetzung der Menschlichen Nehrungs-und-Genussmittel", by Dr. Joseph Koenig, Vol. 2, page 378, and also Chemistry of Coffee, by Dr. Ernest Lockhart, of the Coffee Brewing Institute of America).

The scope of the invention will be fully understood from the description set forth in the following examples:—

EXAMPLE 1

I disintegrate a batch of raw coffee beans into a homogeneous mass and by the known in the industry method of extraction of soluble constituents from vegetable matters, I extract the water soluble constituents of the disintegrated coffee beans.

After the insoluble parts of the disintegrated coffee beans are separated from the resultant solution of the water soluble constituents of the coffee beans, I introduce into the solution as catalyst an inorganic oxidizing compound, by dry weight of the tannin compound in the solution preferably some 0.01 to 0.03% of potassium permanganate ($KMn_2O_4$) and expose the solution for oxidation in an oxygeneous atmosphere even to the action of the oxygen of the air, under a constant heating at a temperature below the boiling point of water, but not lower than 30° C. (84° F.) until by the determination of the qualified tester the tannin compounds in the solution are oxidized to the required degree.

The period of time the solution is to be exposed for oxidation and the percenage of the applied catalyst may be increased or decreased depending on the composition and the tannin contents of every individual batch of coffee beans and the desired degree of oxidation.

To reduce the solution to the required degree of consistency and to eliminate the excess water in the solution, I submit it to evaporation preferably under vacuum, until the concentrate attains the consistency required for economical spray drying the concentrate into a powder.

The formed concentrate I spray dry into a powder under the lowest technically admissible temperatures, required for converting concentrates into dry powder, carrying out the spray drying in a way that no chemical or physical changes in the solids of the formed concentrate can take place. I obtained perfect results by spray drying at a temperature when the outlet temperature was of some 55 to 60° C., and in this way no physical or chemical changes in the powdered extract were noticed.

The operative cooling of the solids particles during the spray drying provides the assurance that no temperature which may cause any chemical or physical change in the powdered dry extract may result. I even applied more adequate additional cooling of the solids which I accomplished by admitting atmospheric or conditioned air directly into the spray drying chamber through a cold side inlet, and established that no reducing sugar and no caffeol were formed.

Only after the so chemically and physically unchanged coffee extract in dry powdery form is produced, I expose the same to action of high degree temperature, required for roasting of coffee and its derivatives, until the required degree of chemical and physical changes were completed, and when most of the sucrose in the powder is converted into reducing sugar, caffeol is formed, and the desired degree of flavour and aroma are developed, much superior to any brand of instant coffee ever produced.

The instant coffee produced by my new above described method, after development the aroma and flavour, does not undergo any further processing and is filled directly into closed containers, therefore the developed flavour and aroma remain in the produced instant coffee for good.

EXAMPLE 2

I place into a container a batch of raw green coffee beans, and cover these beans with boiling water to soak them until they are thoroughly drenched. These drenched coffee beans I comminute into a slurry mass, and by the industry known ways, I extract the water soluble constituents of the disintegrated coffee beans.

After the insoluble parts of the beans are separated from the solution, I introduce into the resulted solution as catalyst an oxidizing inorganic matter in minute quantites like 1.01 to 0.03% by the dry weight of the tannin in the solution.

This solution with the added catalyst, I expose for oxidation to the action of oxygen or the oxygen of the air, under a constant heating under a temperature not lower than 30° C. until the tannin compounds in the solution become oxidized in the desired degree.

To reduce the solution to the desired degree of concentration I submit it to evaporation, preferably under vacuum, until the concen-

trate attains the consistency required for economical spray drying into a powder.

I have ascertained that in spite that the formation of flavour aroma in coffee products is formed mostly during the process of roasting of the powdered soluble constituents of the extracted coffee beans. In some batches certain volatile substances of the raw coffee beans in solution may be evaporated together with the initially evaporated 3—4% of the water in the solution.

In order to save these volatile substances, I recover the initially evaporated 3 to 4% of water, which contains the distilled with the water volatile matter, and continue the evaporation until the required for spray drying consistency of the concentrate is attained, and then I restore to final concentrate the initially evaporated water mixed with the distilled volatile substances.

The formed concentrate I submit to spray drying under technically admissible low temperture, required for converting wet concentrates into dry powders, but in no case under any temperature which may cause any chemical or physical changes in the extracted coffee solids.

To perform the roasting and bound with this roasting chemical and physical changes in the produced by spray drying under low temperature I submit it to the action of high temperature under conditions described in Example 1.

## Example 3

To produce an improved instant coffee from already roasted coffee beans, I extract the water soluble constituents of such coffee beans, previously already roasted. The extraction may be performed by any of the industry known methods of extraction of coffee beans.

After separating the insoluble parts from the formed solution, I introduce into the solution described in Examples 1 and 2 the inorganic oxidizing catalyst, and expose the solution for oxidation to the action of oxygen, preferably of the oxygen of the air, under constant heating at temperatures not lower than 30° C. until by the determination of the qualified tester, the tannin in the solution is sufficiently oxidized.

The percentage of the applied catalyst may be increased or decreased depending on the composition and the tannin content of every individual batch of coffee beans and the desired degree of oxidation.

I have also performed experimental work on oxidation of the extracted water soluble constituents of roasted coffee beans without applying of an oxidizing catalyst and exposed the solution under constant heating to the action of oxygen of the air or in any other oxygeneous atmosphere for a longer time, but the final product as result of over-exposing to heat or to the action of oxygen, the flavour and aroma became deteriorated.

After the oxidation was performed, I submit the solution to evaporation until the required for spray drying consistency is attained.

To convert the formed concentrate of the extracted water soluble constituents of roasted coffee beans into powder, I apply the usual for spray drying high temperatures, generally applied by the coffee industry by spray drying of the extracted water soluble constituents of roasted coffee beans into a dry-powdered state.

## Example 4

In variation of the way of oxidation of the extracted water-soluble solids of coffee beans, as described in Examples 1, 2, 3. I do not introduce the oxidizing catalyst into the formed solution, but as soon as the extraction of the solids was performed, and the insoluble parts of the coffee beans are separated from the solution, I submit the solution at once to evaporation of the excess water until the required for spray drying concentration is attained.

Only after such concentrate is formed, I introduce into the formed concentrate the described in 1, 2 and 3 inorganic oxidizing catalyst, preferably potassium permanganate in a proportion of 0.01 to 0.03% by dry weight of the tannin compounds in the concentrate, and expose the concentrate for oxidation to the action of oxygen, preferably of the oxygen of the air, under constant heating under temperature higher than 30° C. until by the determination of the qualified tester the tannin compounds in the concentrate is oxidized in the desired degree.

The so produced concentrate, with the tannin compounds in it, dully oxidized, I submit to spray drying under temperatures described in the Examples 1, 2 and 3.

My experiments in oxidizing the tannin compounds of coffee beans in solution or concentrate without applying an inorganic oxidizing catalyst, by exposing the extracted solids for oxidation in an oxygeneous atmosphere under constant heating, produced an unhomogeneous instant coffee hardly acceptable for marketing.

RAMON PERECH.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1960.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained